


FORM PTO-110 (REV. 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER KEL01-P111	
<b>TRANSMITTAL LETTER TO THE UNITED STATES          DESIGNATED/ELECTED OFFICE (DO/EO/US)          CONCERNING A FILING UNDER 35 U.S.C. 371</b>				U.S. APPLICATION NO. 09/785644 <b>09/856445</b>	
INTERNATIONAL APPLICATION NO. PCT/EP99/09146		INTERNATIONAL FILING DATE November 22, 1999		PRIORITY DATE CLAIMED November 21, 1998	
TITLE OF INVENTION <b>A THERMOREVERSIBLE FOOD PRODUCT</b>					
APPLICANT(S) FOR DO/EO/US <b>Elizabeth Devine and Conor Ward</b>					
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:					
1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371. 3. <input type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below. 4. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31). 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) a. <input checked="" type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau). b. <input checked="" type="checkbox"/> has been communicated by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)). a. <input type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). 7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input checked="" type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). 9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(3)).					
<b>Items 11 to 20 below concern document(s) or information included:</b>					
11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825. 18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). 19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4). 20. <input type="checkbox"/> Other items or information:					

U.S. APPLICATION NO. <b>097/856445</b> INTERNATIONAL APPLICATION NO. <b>PCT/EP99/09146</b>	ATTORNEY'S DOCKET NUMBER <b>KEK01-P111</b>															
21. <input checked="" type="checkbox"/> The following fees are submitted: <b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):</b> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1000.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$860.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$710.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$690.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00 <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b> \$ 860.00																
Surcharge of \$130.00 for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492(e)). <input type="checkbox"/> 20 <input type="checkbox"/> 30 \$																
<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 20%;">CLAIMS</th> <th style="width: 20%;">NUMBER FILED</th> <th style="width: 20%;">NUMBER EXTRA</th> <th style="width: 20%;">RATE</th> <th style="width: 20%;">\$</th> </tr> </thead> <tbody> <tr> <td>Total claims</td> <td>20 - 20 =</td> <td>-0-</td> <td>x \$18.00</td> <td>\$</td> </tr> <tr> <td>Independent claims</td> <td>2 - 3 =</td> <td>-0-</td> <td>x \$80.00</td> <td>\$</td> </tr> </tbody> </table>	CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	Total claims	20 - 20 =	-0-	x \$18.00	\$	Independent claims	2 - 3 =	-0-	x \$80.00	\$	MULTIPLE DEPENDENT CLAIM(S) (if applicable) + \$270.00 \$
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$												
Total claims	20 - 20 =	-0-	x \$18.00	\$												
Independent claims	2 - 3 =	-0-	x \$80.00	\$												
<b>TOTAL OF ABOVE CALCULATIONS =</b> \$																
<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2. \$ 430.00																
<b>SUBTOTAL =</b> \$																
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)). \$																
<b>TOTAL NATIONAL FEE =</b> \$																
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property + \$																
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a. <input checked="" type="checkbox"/> A check in the amount of \$ 430.00 to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>22-0190</u> . A duplicate copy of this sheet is enclosed. d. <input type="checkbox"/> Fees are to be charged to a credit card. <b>WARNING:</b> Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.																
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.																
SEND ALL CORRESPONDENCE TO Matthew L. Goska Van Dyke, Gardner, Linn & Burkhart, LLP 2851 Charlevoix Drive, S.E. Suite 207 Grand Rapids, MI 49546 Telephone: 616-975-5500																
<div style="text-align: right;">           SIGNATURE  <u>Matthew L. Goska</u>          NAME          42 594          REGISTRATION NUMBER       </div>																

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JCO3 Rec'd PGT/STC

21 MAY 2001

PATENT  
KEL01-P111

Express Mail No. EL846001905US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s) : ELIZABETH DEVINE and CONOR WARD

For : A THERMOREVERSIBLE FOOD PRODUCT

BOX PATENT APPLICATION

Commissioner of Patents and Trademarks  
Washington, D.C. 20231

Dear Sir:

PRELIMINARY AMENDMENT

Prior to examination and calculation of the filing fee, please amend claims 1, 3-7, 9 and 10 so that they appear as follows:

-1- (Amended)

A food product having a top layer comprising less than 70% (w/w) water and an amount of at least one top thermoreversible setting agent sufficient to maintain the top layer in a gel phase at room temperature, whilst permitting thermoreversion to a liquid phase at a temperature elevated above room temperature, with the proviso that sodium caseinate is substantially absent

-3- (Amended)

A food product according to Claim 2, in which the thermoreversible setting agent is selected from the group consisting of gelatine, agar, gellan gum, methyl celluloses, guar gum, at least one carrageenan, gum arabic, xanthan gum, locust bean gum or mixtures thereof; and the top emulsifier is selected from the group consisting of monoglycerides, di-glycerides, sucrose esters; or mixtures thereof.

-4- (Amended)

A food product according to Claim 3, in which the top layer contains 0.1 to 1.75% (w/w) of the at least one top thermoreversible setting agent, selected from the group consisting of gelatine, carrageenan or mixtures thereof.

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-5- (Amended)

A food product according to Claim 1, in which a base layer contains 0.1 to 3.5% (w/w) of at least one base thermoreversible setting agent, the base thermoreversible setting agent being selected from the group consisting of gelatine, agar, gellan gum, methyl celluloses, guar gum, at least one carrageenan, gum arabic, xanthan gum, locust bean gum or mixtures thereto.

-6- (Amended)

A food product according to Claim 1, in which the top layer contains 40-65% (w/w) water.

-7- (Amended)

A food product according to Claim 1, in which the top layer contains 20-40% fat.

-9- (Amended)

A food product according to Claim 1 in which the top layer contains at least one comestibly suitable alcohol.

-10- (Amended)

A food product according to Claim 8 in the form of a cream liqueur.

Please add new claims 12 -20 as follows:

-12- (New)

A food product according to Claim 3, in which the top layer contains 0.1 to 1.75% (w/w) of the least one thermoreversible setting agent selected from the group consisting of gelatine, kappa-carrageenan, iota-carrageenan, mixtures of kappa- and iota-carrageenan; and mixtures of one or both of kappa and iota-carrageenan with gelatine.

-13- (New)

A food product according to Claim 1, in which a base layer contains 0.1 to 3.5% (w/w) of at least one base thermoreversible setting agent, the base thermoreversible setting agent being selected from the group consisting of gelatine, carrageenan and mixtures thereof.

-14- (New)

A food product according to claim 1, in which a base layer contains 0.1 to 3.5% (w/w) of at least one base thermoreversible setting agent, the base thermoreversible setting agent being selected from the group consisting of gelatine, kappa-carrageenan, iota-carrageenan, mixtures of kappa- and iota- carrageenan and mixtures of one or both of kappa and iota-carrageenan with gelatine.

-15- (New)

A food product according to Claim 1, in which the top layer contains 40-50% (w/w) water.

-16- (New)

A food product according to claim 2, in which the top layer contains 40-50% (w/w) water.

-17- (New)

A food product according to Claim 4, in which the top layer contains 20-40% (w/w) fat.

-18-(New)

A food product according to Claim 2, in which the top layer contains at least one comestibly suitable alcohol.

-19- (New)

A food product according to Claim 4, in which the top layer contains at least one comestibly suitable alcohol.

-20-(New)

A food product according to Claim 9, in the form of a cream liqueur.

#### REMARKS

The foregoing amendments are being made to remove multiple dependencies, correct typos, and better conform the claims to U.S. claim practice. None of the amendments are being made for purposes of patentability. A copy of the amendments made are attached on the following sheet entitled "Version With Markings to Show Changes Made."

Respectfully submitted,

ELIZABETH DEVINE and CONOR WARD

By: Van Dyke, Gardner, Linn & Burkhart, LLP

Dated: May 21, 2001

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Grand Rapids, Michigan 49588-8695  
(616) 975-5500

VERSION WITH MARKINGS TO SHOW CHANGES MADE

-1- (Amended)

A food product having a top layer comprising less [then] than 70% (w/w) water and amount of at least one top thermoreversible setting agent sufficient to maintain the top layer in a gel phase at room temperature, whilst permitting thermoreversion to a liquid phase at a temperature elevated above room temperature, with the proviso that sodium caseinate is substantially absent

-3- (Amended)

A food product according to Claim 2, in which the thermoreversible setting agent is selected from the group [comprising] consisting of gelatine, agar, gellan gum, methyl celluloses, guar gum, at least one carrageenan, gum arabic, xanthan gum, locust bean gum or mixtures thereof; and the top emulsifier is selected from the group consisting of monoglycerides, diglycerides, sucrose esters; or mixtures thereof.

-4- (Amended)

A food product according to Claim 3, in which the top layer contains 0.1 to 1.75% (w/w) of the at least one top thermoreversible setting agent, the base thermoreversible setting agent [preferably] selected from the group [comprising] consisting of gelatine, carrageenan or mixtures thereof, [most preferably selected from the group comprising gelatine, kappacarrageenan, iota-carrageenan, mixtures of kappa- and iota-carrageenan; and mixtures of one or both of kappa and iota-carrageenan with gelatine.]

-5- (Amended)

A food product according to [any one of the preceding claims] Claim 1, in which a base layer contains 0.1 to 3.5% (w/w) of at least one base thermoreversible setting agent, the base thermoreversible setting agent being selected from the group consisting of [comprising] gelatine, agar, gellan gum, methyl celluloses, guar gum, at least one carrageenan, gum arabic, xanthan gum, locust bean gum or mixtures thereof, [, more preferably selected from gelatine, carrageenan and mixtures thereof, most preferably selected from gelatine, kappa-carrageenan, iota-carrageenan, mixtures of kappa- and iota-carrageenan and mixtures of one or both of kappa and iota-carrageenan with gelatine.]

-6- (Amended)

A food product according to Claim 1 [any one of the preceding claims], in which the top layer contains 40-65% (w/w) water[, preferably 40-50% (w/w) water].

-7- (Amended)

A food product according to Claim 1, [any one of the preceding claims] in which the top layer contains 20-40% fat.

-9- (Amended)

A food product according to Claim 1 [any one of the preceding claims], in which the top layer contains at least one comestibly suitable alcohol.

-10- (Amended)

A food product according to Claim 8 [or 9] in the form of a cream liqueur.

A THERMOREVERSIBLE FOOD PRODUCT

5

The present invention concerns a thermoreversible food product.

10 The term "thermoreversible" is intended to mean that which is convertible from a gel phase at room temperature to a liquid phase at a temperature elevated above room temperature. The term "thermoreversible" also includes the preferred term "thermointerchangeable" as that which is convertible  
15 from a gel phase at room temperature to a liquid phase at a temperature elevated above room temperature and is reconvertible back to a gel phase when cooled to room temperature.

20 It is an object of the present invention to provide a food product which is gelled at room temperature and which, following heating in a microwave, has completely melted.

25 It is a further object of the present invention to provide a bilayered food product in which both layers are gelled at room temperature and in which, following heating in a microwave, both layers are completely melted, but remain as substantially separate liquid  
30 layers.

It is a still further object of the present invention to provide a bilayered food product in which colour

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migration between the two layers is substantially eliminated, after the food product has been microwaved.

It is also an object of the present invention to  
5 provide a multilayered food product, in which all layers are gelled at room temperature and in which, following heating in a microwave, at least two of the layers are completely melted, but remain substantially separate liquid layers, and in which colour migration,  
10 between those layers, is substantially eliminated.

The food product may be, for example, a coffee liqueur product, having a base layer containing coffee and alcohol and having a top layer containing an animal fat  
15 or a mixture thereof, a vegetable fat or a mixture thereof or a mixture of animal and vegetable fats. It is envisaged that both layers of such a food product may be melted to their respective liquid phases and heated by means of a microwave.

20 In a first embodiment, the invention provides a food product having a top layer comprising less than 70%, preferably less than 65%, more preferably less than 60%, most preferably less than 50%, (w/w) water and an  
25 amount of at least one top thermoreversible setting agent sufficient to maintain the top layer in a gel phase at room temperature whilst permitting thermoreversion to a liquid phase at a temperature elevated above room temperature, with the proviso that  
30 sodium caseinate is substantially absent.

Preferably, the top layer contains 40-65% (w/w) water, more preferably 40-50% (w/w) water.

More preferably, the top layer contains at least 40% (w/w) water.

- 5 By "room temperature", we mean at or below 23°C. By "temperature elevated above room temperature", we mean that the temperature in the relevant layer is, following heating by microwave, at or above 40°C, preferably at or above 45°C, more preferably at or  
10 above 50°C, most preferably at or above 55°C.

- Preferably, the food product additionally includes a base layer comprising at least one base thermoreversible setting agent in an amount sufficient  
15 to maintain the base layer in a gel phase at room temperature, whilst permitting thermoreversion to a liquid phase at a temperature elevated above room temperature.

- 20 Advantageously, the top layer additionally comprises at least one top emulsifier and more than 15% (w/w) fat. More advantageously, the top layer contains more than 20% (w/w) fat and most advantageously 20-40% (w/w) fat. The fat can be of animal or vegetable origin, or a  
25 mixture thereof.

- Preferably, the at least one top thermoreversible setting agent is combined with the at least one top emulsifier selected from at least one mono-glyceride of  
30 a fatty acid or mixtures thereof; at least one di-glyceride of a fatty acid or mixtures thereof; at least one sucrose ester or mixtures thereof; or mixtures of two or more of mono-glycerides, di-glycerides and

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sucrose esters. More preferably, the top layer contains 0.1 to 1.75% (w/w) of the at least one top thermoreversible setting agent. Even more preferably, the base layer contains 0.1 to 3.5% (w/w) of the at least one base thermoreversible setting agent.

- In a conventional cream liqueur system, sodium caseinate is used as an emulsifier and as a stabiliser. However, in a microwavable food product, which is the subject of the present invention, the co-presence of sodium caseinate and alcohol causes skin formation when the food product is microwaved. It was, therefore, necessary to preclude the use of sodium caseinate.
- It is believed that the present invention overcomes the problem of colour migration by providing a top layer having less than 70% (w/w) water - this reduces hydrogen ion migration, which is accompanied by colour migration, from the base layer to the top layer, which hydrogen ion migration affects the pH of the top layer and may also be accompanied by a breakdown in the stability of the top layer's emulsion, causing it to lose its thermoreversibility.
- Suitable emulsifiers should not form a skin on the upper surface of the top layer when the top layer is heated - it is thought that such skin formation is related to denaturation of a proteinaceous emulsifier, so that such emulsifiers should be avoided. Suitable emulsifiers must be comestible and include, but are not limited to, mono-glycerides of fatty acids, di-glycerides of fatty acids, sucrose esters or mixtures

thereof. Sucrose esters endow good stability, but their organoleptic properties need to be improved.

Suitable top and base thermoreversible setting agents must be comestible and include, but are not limited to, gelatine, agar, gellan gum, methyl celluloses, guar gum, carrageenan (preferably kappa and/or iota carrageenan), gum arabic, xanthan gum, locust bean gum or mixtures thereof. When the thermoreversible setting agent is gelatine, the top layer may contain 0.75 to 1.75% (w/w), more preferably 1.0 to 1.5% (w/w) gelatine and the base layer may contain 1.5 to 3.5% (w/w), preferably 2.0 to 3.25% (w/w), more preferably 2.25 to 2.75% (w/w) gelatine. When the thermoreversible setting agent is carrageenan, the top layer may contain 0.1 to 1.0% (w/w), preferably 0.25 to 0.75% (w/w) carrageenan and the base layer may contain 0.1 to 1.5% (w/w), preferably 0.2 to 0.75% (w/w) carrageenan. If a mixture of carrageenans is used, they may be present in a ratio of 2:1 to 1:2, preferably 1.5:1 to 1:1.5 (ratio of kappa to iota carrageenan).

Gelatine is obtained from collagen found in animal tissues, including the skin, tendons and ossein of bones.

In a second embodiment, the invention provides a cream liqueur food product having a top layer comprising (i) less than 70% (w/w) water; (ii) more than 15% (w/w) fat; (iii) at least one top emulsifier; and (iv) at least one top thermoreversible setting agent, the at least one top emulsifier and the at least one thermoreversible setting agent being sufficient to

maintain the top layer in a gel phase at room temperature, whilst permitting thermoreversion to a liquid phase at a temperature elevated above room temperature, with the proviso that the, or each, top  
5 emulsifier or setting agent is not sodium caseinate.

The invention also provides a method for forming a layer of a food product, comprising at least one thermoreversible setting agent and/or at least one  
10 emulsifier, the method comprising the steps of providing a first liquid phase at a temperature below room temperature; providing a second liquid phase comprising an amount of at least one setting agent and/or at least one emulsifier sufficient to maintain a  
15 mixture of the first and second liquid phases, when mixed in a thermoreversible gel phase at room temperature with the proviso that the, or each, setting agent and/or emulsifier is not sodium caseinate, the second liquid phase being at a temperature raised above  
20 room temperature; and mixing the first and second liquid phases to obtain the top layer.

By "raised temperature" is meant a temperature at or above 30°C, preferably at or above 35°C.  
25

It will be appreciated that, if the food product comprises a top layer and a base layer, the aforementioned method comprises the steps of providing first and second base phases; mixing the first and  
30 second base phases and allowing the thus mixed first and second base phases to set; providing first and second top phases; mixing the first and second top

phases and allowing the thus mixed first and second top phases to set on the base layer.

According to a further embodiment of the invention  
5 there is provided a method for forming a layer of a food product comprising at least one thermoreversible setting agent and/or at least one emulsifier sufficient to maintain the layer in a gel phase with the proviso that the, or each, setting agent and/or emulsifier is  
10 not sodium caseinate.

It will be appreciated that, if the food product comprises a top layer and a base layer, the  
aforementioned method comprises the steps of providing  
15 the first layer, allowing the first layer to set; providing the second layer and allowing the second layer to set on the base layer.

Advantageously, the food product is a cream liqueur.  
20 By "cream liqueur" is meant a bi- or multi-layered beverage whose top layer contains fat of animal and/or vegetable origin and at least one of whose other layers contains alcohol. If the cream liqueur is bilayered, the volume ratios of the top and base layer should be  
25 in the range 1:1 to 1:12, preferably 1:1 to 1:10, most preferably about 1:2. Hereinafter is exemplified a cream liqueur food product in the form of an "Irish Coffee" having two layers, namely a base layer and the top layer. It will, of course, be appreciated that the  
30 cream liqueur food product may, alternatively, be a "French Coffee" if brandy is added instead of Irish whiskey in the base layer. Similarly, a "Russian Coffee" (vodka) or "Caribbean Coffee" (rum), etc., are

also envisaged. The present invention is not of course limited to coffee-containing cream liqueur food products (coffee cream liqueur food products)

- 5 Any comestibly suitable alcohol in the base and/or top layer may be selected from spirits, such as neutral spirit, whiskey, whisky, brandy, vodka, rum or a mixture thereof. Preferably, the base layer contains 7 to 20% (w/w), preferably 8 to 12.5% (w/w), more preferably 8.75 to 10% (w/w) alcohol. More preferably, the top layer contains 7 to 20% (w/w), preferably 8 to 12.5% (w/w), more preferably 8.75 to 10% (w/w) alcohol. Advantageously, the cream liqueur food product contains 7 to 20% (w/w), most preferably 8.75 to 10.5% (w/w) alcohol.

The invention will now be described and exemplified in the following examples, which are not intended to limit the scope of the invention.

#### Example 1

A gelatine-containing cream liqueur food product is prepared in the following manner.

- 25 The base layer is made first and needs to set before the top layer is applied. In this example, the base layer is made by forming first and second liquid base phases, the first liquid base phase being a coffee flavoured solution and the second liquid base phase being a 7.5% (w/w) gelatine solution.

Table 1 shows the ingredient list for the base and top layers of two formulations of the present invention -

Formulations A and B - each of which is similarly prepared.

Table 1

BASE LAYER - INGREDIENT LIST		
Ingredients	A % (w/w)	B % (w/w)
Water	72.34	72.14
Sugar	15.00	15.00
Gelatine-Sherex***	2.45	2.45
Caramel (Quest 15752)	0.49	0.49
Neutral Spirit	7.64	7.75
Whiskey	1.13	1.13
Flavour	0.39*	0.49**
4% NaOH Solution	0.55	0.55

\*Coffee (Quest 2A92529)

\*\*Coffee QL 15052

\*\*\*Sherex is a 240-bloom pork gelatine supplied by Quest International.

10

TOP LAYER - INGREDIENT LIST		
Ingredients	A % (w/w)	B % (w/w)
Water	46.75	42.44
Double Cream	17.94	18.53
Akolat* (Trade Mark of Karlshamns) B15	13.70	14.15
Sugar	9.13	11.89
Neutral Spirit	7.85	8.11
Whiskey	1.17	1.21
Starch - Thermtex	0.91	-
Tri Sodium Citrate	0.08	0.08
Gelatine - Sherex	1.37	1.42
E471 Emulsifier (Admul MG 4223)**	0.18	0.19
Flavour (Cocoa DA06008)	0.91	1.89
Whisky - nn 11034	-	0.09

\*A vegetable fat, specifically, a nonlauric speciality fat based on a blend of refined and partly hydrogenated oils and fats and containing emulsifiers (E471 and E322) and natural



and/or near nature identical butter fats. Akolat 15 can be used in a large number of applications in which butter fat is normally used.

- 5 \*\*Admul MG 4223 is a mono-diglyceride prepared from edible vegetable oil and is recommended for use as a water-in-oil emulsifier in table margarine; as an aerating agent and shelf-life extender in bakery margarines and shortenings; and as an aerating agent in ice-cream and imitation creams.

- 10 Formulation B has a slightly higher alcohol content at 10.1% than Formulation A at 9.97%, in their respective base layers - this improves its organoleptic properties and, possibly, its microbial stability. In the  
15 respective top layers, sugar and flavour contents are higher in Formulation B, than in Formulation A, to improve organoleptic properties. These changes, in turn, affected the weights of the respective top layers which, then, affects the respective % (w/w).

- 20 The first liquid base phase is prepared by heating 30% of the water used to make the base to 15°C in a jacketed insulated tank equipped with a mixer and dissolving the sugar therein.

- 25 The caramel, neutral spirit, whiskey and coffee extract flavour are then added and the solution corrected to pH 6-7 by adding 4% NaOH. When fully dissolved, 25.1% of the water used to make the base is added as ice,  
30 thereby chilling the solution to 4°C.

- The second liquid base phase is prepared by using 44.9% of the water used to make the base so as to make a 7.5% (w/w) gelatine solution. The water is heated to 70°C  
35 in a jacketed insulated vessel equipped with a stirrer and the gelatine powder is added under agitation. The solution is maintained at 70°C for 3 minutes, in order

to ensure that the gelatine is solubilised. The mixture is then cooled, with stirring to 35°C.

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The base layer is then obtained by transferring two  
5 volumes of the first liquid base phase into the end  
mould, for example, a glass and, thereafter, dispensing  
one volume of the gelatine solution (second liquid base  
phase) into the mould in such a way as to ensure that  
the first and second liquid base phases are completely  
10 mixed. This is achieved using twin piston volumetric  
fillers, which are equipped with non-drip filling  
nozzles and individually jacketed hoppers (at 4°C and  
at 35°C, respectively) for the first and second liquid  
base phases. The second liquid base phase hopper  
15 should be a low volume tank, to ensure rapid turnover  
and short holding times of the second liquid base phase  
and its filling head, down to the filling nozzle,  
should be trace heated to 35°C. Both filling  
heads/mechanisms should be attemperated by initially  
20 re-circulating the respective first and second liquid  
base phases. 66mls of the first liquid base phase (at  
4°C) and 33mls of the second liquid base phase (at  
35°C) are mixed by simultaneously filling into a 17cl  
"tulip" glass with a resulting temperature, when mixed,  
25 of 16°C approximately. Filling is through twin off-  
centred, non-drip filling nozzles, to ensure adequate  
mixing.

Extensive testing of a wide variety of glass types has  
30 demonstrated that the use of a "tulip" glass is  
preferred for manufacturing the product and for  
microwaving it prior to consumption. By "tulip" glass,  
is meant a glass of substantially tulip shape.

- The "tulip" type glass is the most stable on the production line, it maximises the mixing of the two phases at the fillers and shows good load-bearing characteristics for foiling. On microwaving, the "tulip" glass does not develop convection currents and therefore does not display the mixing of the two layers which is characteristic of other glass shapes.
- 10 At the optimum temperature of 4°C for the first liquid base phase and 35°C for the second liquid base phase, the base layer sets in a period of 7-10 minutes at 10°C-20°C air temperature.
- 15 The 17cl glass containing the base layer at approximately 16°C is transferred to a covered, positively-pressured, first accumulation conveyor, whereupon it is retained for 20 minutes so as to ensure a firm set of the base layer.
- 20 The top layer is made in the following manner. Once again, the top layer is formed by providing, separately, first and second liquid top phases which are thereafter mixed.
- 25 The first liquid top phase is made by heating 60.43% of the water to make the topping to 80°C in a jacketed insulated tank equipped with a high speed stirrer, the water being agitated vigorously. Tri-sodium citrate, emulsifier (E471), starch, sugar, Akolat (Trade Mark) B15 vegetable fat, double cream, neutral spirit, whiskey and cream liqueur flavours are then added in that order. The mixture is heated to 55°C and, with
- 30

1  
vigorous stirring, the solution is maintained at 55°C  
for 2 minutes. The solution is then double homogenised  
at 4,000 psi. The first liquid top phase is then  
cooled in-line to 13°C and can be stored at a  
5 temperature of 13°C in a jacketed insulating tank  
equipped with a stirring paddle.

10 The second liquid top phase is prepared by using 39.57%  
of the water used to make the topping so as to make a  
7.5% (w/w) gelatine solution, the water being heated to  
70°C in a jacketed insulated vessel equipped with a  
stirrer and the gelatine powder being added under  
agitation. The solution is maintained at 70°C for 3  
minutes, so as to ensure that the gelatine fully  
15 solubilised. The mixture is then cooled, with  
stirring, as rapidly as is possible to 35°C.

20 The top layer is then obtained by mixing the first and  
second liquid phases thoroughly in a ratio of 4.0 parts  
by volume of first liquid top phase and 1.0 parts by  
volume of second liquid top phase. Specifically, twin  
piston volumetric fillers are used, each equipped with  
individually jacketed hoppers (at 13°C and at 35°C,  
respectively) for the first and second liquid top  
25 phases.

The filler is equipped with a single filling and mixing  
head to thoroughly mix the discharges of both pistons  
before their deposition in the 17cl glass which  
30 contains the set base layer. Filling volumes of 40ml  
and 10ml of first and second liquid top phases,  
respectively, are used, the filling being via a single  
centred non-drip filling nozzle.

Once again, the second liquid top phase hopper should be a low volume tank to ensure rapid turnover and short holding times of the second liquid top phase. Its filling head, down to the mixing head, should be trace heated to 35°C. As before, both filling heads/mechanisms should be attemperated by initially re-circulating their respective products. The top layer takes 8-10 minutes to set at an ambient temperature of 10°-20°C.

The 17cl glass containing the set base layer and the 50ml top layer is then passed to a second accumulation conveyer (covered and positively air pressurised) for a 20 minute residence time until a firm set has been achieved. The 17cl "tulip" glass containing the set base layer and the set top layer is then transferred to a rotary foiling station where a microwavable foil is picked from a magazine, placed on the glass rim and conduction sealed at 180°C for 0.5-1.5 seconds. The heated sealing head should be a rubber/metal composite and be fixed on flexible mountings.

The foil should be heavy gauge aluminium with no rough edges to minimise the risk of "arcing" in the microwave. The foil underseal is specifically formulated to seal to the very small cross section of the glass rim within the time/temperature allowed.

The product is then passed for packaging.

Experiments have shown that it is possible to make a cream liqueur food product with a stable cream-

containing top layer with a shelf life of up to 12 weeks at room temperature i.e., less than 23°C, without using sodium caseinate to stabilise the emulsion.

- 5 The presence of 0.18% (or 0.19%) (w/w) E471, as emulsifier, in the top layer, yields a top layer with good organoleptic properties which does not form a skin after microwaving.
- 10 It will be appreciated that the temperature and residence times indicated herein are ambient temperature dependent and may be varied, depending on the actual ambient temperature experienced.
- 15 When it is desired to consume the food product, the glass containing the base layer and top layer and covered with the foil is placed in a 700W microwave oven for 1 minute to heat the top layer to approximately 52°C and the base layer to approximately
- 20 62°C. It will, of course, be appreciated that the microwave conditions will vary, depending on the make, age and wattage of the microwave oven.

#### **Example 2**

- 25 A carrageenan-containing cream liqueur food product is made in the following manner.

The base layer is made first and needs to set before the top layer is applied. The base layer comprises of

30 a coffee flavoured solution with neutral spirit, whiskey, minerals (calcium chloride dihydrate), sugar and a thermoreversible setting agent. The top layer comprises of a flavoured solution of animal and

vegetable fats, neutral spirit and whiskey, emulsifiers and two thermoreversible setting agents.

Table 2 shows the ingredients for the base and top layer.

Table 2

BASE LAYER - INGREDIENT LIST	
Ingredients	% (w/w)
Water	74.78
Sugar	15.00
Deltagel 379*	0.39
Quest Caramel 15752	0.49
Neutral Spirit	7.64
Whiskey - Bushmills	1.13
Quest Coffee 2A92529	0.39
Calcium Chloride Dihydrate	0.17

\* Deltagel 379 is kappa carrageenan for use in food products and is supplied by Quest International Ireland Limited.

TOP LAYER - INGREDIENT LIST	
Ingredients	% (w/w)
Water	42.78
Sugar	11.99
Akolat B15* - Karlshamns	14.27
Cream	18.69
Neutral Spirit	8.18
Whiskey - Bushmills	1.22
Cocoa Flavour DA06008	1.90
Admul MG4223 (emulsifier) (E471)	0.19
Whiskey NN11034	0.10
Deltagel 388**	0.30
Calcium Chloride Dihydrate	0.17
Deltagel 379*	0.20

\* Deltagel 379 is kappa carrageenan for use in food products and is supplied by Quest International Ireland Limited.

\*\*Deltagel P388 is iota carrageenan for use in food products and containing carrageenan (E407).

The base layer is prepared by heating 90% of the water to 100°C in a tank equipped with a mixer and heating

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and cooling jackets. Calcium chloride dihydrate is added achieving a temperature of 90°C, followed by the sugar and the thermoreversible setting agent (Deltagel 379) which have been blended prior to this. Mixing is continued at 80-90°C (15-20 mins approx.) until the solution is clear. It is then chilled to 45-50°C - but never below this temperature range. The spirits are diluted with the remainder of the water content (10%) and are then added with colours and flavours to the tank.

The base layer is then obtained by filling a volume of the liquid base into an end mould, for example, a glass. 100ml of the liquid base layer (at 45-50°C) is filled into a 17cl "tulip" glass. The filler hopper for the liquid base should be a low volume tank, to ensure rapid turnover and short holding times and its filling head, down to the filling nozzle, should be trace heated to 45-50°C. The filling head/mechanism should be attempered by initially re-circulating the respective solution.

As in Example 1, a "tulip" glass is preferred for manufacturing the product and for microwaving it prior to consumption.

The 17cl glass containing the base layer at approximately 40°C is transferred to a covered, positively-pressured, first accumulation conveyor, whereupon it is retained for up to 25 minutes (air temperature 10-20°C) so as to ensure a firm set of the base layer.





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foiling station where a microwavable foil is picked from a magazine, placed on the glass rim and conduction sealed at 180°C for 0.5-1.5 seconds. The heated sealing head should be a rubber/metal composite and be  
5 fixed on flexible mountings.

As in Example 1, the foil should be heavy gauge aluminium with no rough edges to minimise the risk of "arcing" in the microwave. The foil underseal is  
10 specifically formulated to seal to the very small cross section of the glass rim within the time/temperature allowed.

Experiments have shown that it is possible to make an  
15 alcohol-containing food product with a stable cream-containing top layer with a shelf life of up to 12 weeks at temperatures up to 42°C, without using sodium caseinate to stabilise the emulsion.

20 The presence of 0.19% (w/w) E471, as emulsifier, in the top layer, yields a top layer with good organoleptic properties which does not form a skin after microwaving.

25 It will be appreciated that the temperature and residence times indicated herein are ambient temperature dependent and may be varied, depending on the actual ambient temperature experienced.

30 When it is desired to consume the cream liqueur food product, the glass containing the base layer and top layer and covered with the foil is placed in a 800W microwave oven for approximately 1 minute 30 secs (half

power) to heat the top and base layers to approximately 65°C. It will, of course, be appreciated that the microwave conditions will vary, depending on the make, age and wattage of the microwave oven.

5

It will be appreciated that, although Example 1 exemplifies a food product containing gelatine as the thermoreversible setting agent and Example 2

10 exemplifies a food product containing carrageenan (base layer) and carrageenans (top layer) as thermoreversible setting agents, it is also within the scope of the present invention to use gelatine (or other thermoreversible setting agent(s)) in one layer and carrageenan(s) (or other thermoreversible setting  
15 agent(s)) in another layer.

**CLAIMS:**

1. A food product having a top layer comprising less  
5 then 70% (w/w) water and an amount of at least one top  
thermoreversible setting agent sufficient to maintain  
the top layer in a gel phase at room temperature,  
whilst permitting thermoreversion to a liquid phase at  
a temperature elevated above room temperature, with the  
10 proviso that sodium caseinate is substantially absent.
2. A food product according to Claim 1, in which the  
top layer additionally comprises more than 15% (w/w)  
fat and at least one top emulsifier.
- 15 3. A food product according to Claim 2, in which the  
thermoreversible setting agent is selected from the  
group comprising gelatine, agar, gellan gum, methyl  
celluloses, guar gum, at least one carrageenan, gum  
20 arabic, xanthan gum, locust bean gum or mixtures  
thereof; and the top emulsifier is selected from mono-  
glycerides, di-glycerides, sucrose esters; or mixtures  
thereof.
- 25 4. A food product according to Claim 3, in which the  
top layer contains 0.1 to 1.75% (w/w) of the at least  
one top thermoreversible setting agent, preferably  
selected from the group comprising gelatine,  
carrageenan or mixtures thereof, most preferably  
30 selected from the group comprising gelatine, kappa-  
carrageenan, iota-carrageenan, mixtures of kappa- and  
iota-carrageenan; and mixtures of one or both of kappa  
and iota-carrageenan with gelatine.

5. A food product according to any one of the preceding claims, in which a base layer contains 0.1 to 3.5% (w/w) of at least one base thermoreversible setting agent, the base thermoreversible setting agent being selected from the group comprising gelatine, agar, gellan gum, methyl celluloses, guar gum, at least one carrageenan, gum arabic, xanthan gum, locust bean gum or mixtures thereof, more preferably selected from gelatine, carrageenan and mixtures thereof, most preferably selected from gelatine, kappa-carrageenan, iota-carrageenan, mixtures of kappa- and iota-carrageenan and mixtures of one or both of kappa and iota-carrageenan with gelatine.
6. A food product according to any one of the preceding claims, in which the top layer contains 40-65% (w/w) water, preferably 40-50% (w/w) water.
7. A food product according to any one of the preceding claims, in which the top layer contains 20-40% (w/w) fat.
8. A food product according to Claim 5, in which the base layer contains at least one comestibly suitable alcohol.
9. A food product according to any one of the preceding claims, in which the top layer contains at least one comestibly suitable alcohol.
10. A food product according to Claim 8 or 9, in the form of a cream liqueur.

11. A cream liqueur food product having a top layer comprising

- (i) less than 70% (w/w) water;
- 5 (ii) more than 15% (w/w) fat;
- (iii) at least one top emulsifier; and
- (iv) at least one top thermoreversible setting agent,

the at least one top emulsifier and the at least one  
10 thermoreversible setting agent being sufficient to maintain the top layer in a gel phase at room temperature, whilst permitting thermoreversion to a liquid phase at a temperature elevated above room temperature, with the proviso that the, or each, top  
15 emulsifier or setting agent is not sodium caseinate.

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### DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor, if only one name is listed below, or an original, first and joint inventor, if plural names are listed below, of the subject matter which is claimed and for which a patent is sought on the invention entitled **A THERMOREVERSIBLE FOOD PRODUCT** the specification of which was filed as an international application on November 22, 1999 and assigned serial no. **PCT/IE 99/09146**.

I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office (the Office), all information which is known by me to be material to patentability as defined in Title 37, Code of Federal Regulations (C.F.R.), § 1.56.

### CLAIM OF PRIORITY

I hereby claim foreign benefits under Title 35, United States Code (U.S.C.), Section 119, or Section 365(b) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

British Patent Application 9825446.9, filed November 21, 1998

### POWER OF ATTORNEY

I hereby appoint the patent law firm of Van Dyke, Gardner, Linn & Burkhart, LLP, 2851 Charlevoix Drive, S.E., Suite 207, Grand Rapids, Michigan 49546, telephone number 616/975-5500, facsimile number 616/975-5505, and the individual patent attorneys and patent agents at such patent law firm, namely, Daniel Van Dyke, Reg. No. 25 046; Donald S. Gardner, Reg. No. 25 975; Terence J. Linn, Reg. No. 30 283; Frederick S. Burkhart, Reg. No. 29 288; Catherine S. Collins, Reg. No. 37 599; Matthew L. Goska, Reg. No. 42 594; and Timothy A. Flory, Reg. No. 42 540, my attorney(s) or agent(s) with full power of substitution and revocation, to prosecute this application and to transact all business in and to receive all correspondence from the Patent and Trademark Office connected therewith.

All statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true, and further, these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001, and that such willful false statements may jeopardize the validity of this application or any patent issued thereon.

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